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Indian Standard

RECOMMENDED METHODS FOR
THE REMOVAL OF NON-FIBROUS MATTER
PRIOR TO QUANTITATIVE ANALYSIS
OF FIBRE MIXTURES

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RECOMMENDED METHODS FOR THE REMOVAL OF NON-FIBROUS MATTER PRIOR TO QUANTITATIVE ANALYSIS OF FIBRE MIXTURES

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Indian Standard

RECOMMENDED METHODS FOR THE REMOVAL OF NON-FIBROUS MATTER PRIOR TO QUANTITATIVE ANALYSIS OF FIBRE MIXTURES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 January 1979, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 It is common practice for various additions to be made to fibres, yarns and fabrics for assisting, the processing and manufacture or modifying the properties of the finished material. These usually result in appreciable increases in mass and often affect the solubility of the fibres. It has also to be borne in mind that fibres generally contain a small proportion of naturally occurring non-fibrous substances. The removal of these non-fibrous substances is therefore necessary before conducting the procedure for quantitative chemical analysis of fibre mixtures given in various Indian Standards.

0.3 The removal of certain types of non-fibrous matter, particularly when more than one substance is present, may demand the exercise of considerable chemical resource, and each material to be treated for removal of its non-fibrous matter should be regarded as an individual problem. The procedures suggested do not pretend to be complete, and it should not be assumed that the procedures described in 5 will have no effect on the physical and chemical properties of the textile materials concerned. Further more, these procedures are only applicable where the non-fibrous substances are known or can be identified with certainty.

0.4 For the purpose of this standard, dyes are not considered as non-fibrous matter but as an integral part of the textile and so they are not mentioned. Some prints are made with resin bonded pigments which cannot be regarded as part of the fibre substance. They involve a greater addition of mass to the fibre than dyes and it would be desirable to remove them, but it is rarely, if ever, possible to do so. Similarly certain finishes cannot be removed. In the present state of knowledge quantitative analysis to the degree of accuracy required cannot be achieved in these cases.

0.5 It may be assumed that Soxhlet extraction under the conditions described in 5 will ensure adequate removal of oils, fats and waxes. With other non-fibrous substances it is necessary, wherever possible, to check that removal is complete.

0.6 This standard is mainly based on ISO/TR 5090-1976 Textiles—Methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes recommended procedures for the removal of certain commonly found types of non-fibrous matter from the fibres. The fibres to which the procedures are applicable are also listed in Table 1.

1.2 Identification of the non-fibrous matter and of the fibres present has not been covered in this standard. In certain cases, the complete elimination of all the added matter like compounds which react with the fibre substrate is impracticable. The quantity remaining should not affect the quantitative analysis. On the other hand it is essential to minimize the chemical degradation of the fibres.

2. TERMINOLOGY

2.1 For the purpose of this standard, non-fibrous matter comprises :

- a) processing aids such as lubricants and sizes (but excludes jute-batching oils), and
- b) naturally occurring non-fibrous substances, but
- c) not such compounds which react with the fibre substrate.

3. PRINCIPLE

3.1 Where possible, non-fibrous matter is removed by a suitable solvent, but in many cases the removal of certain finishes involve some chemical modification of the finish. In addition chemical degradation of the fibre substance cannot always be avoided.

*Rules for rounding off numerical values (revised).

TABLE 1 APPLICABILITY OF PROCEDURES

(*Clauses 1.1 and 5.0*)

NON-FIBROUS MATTER (1)	METHOD (2)	APPLICABLE IN PRESENCE OF (3)	NOT APPLICABLE IN PRESENCE OF (4)
Oils, fats and waxes	5.1	Most fibres	Elastane
Soaking oils	5.2	Nett silk	—
Starch	5.3	Cotton (Note 1) Linen (Note 2) Viscose Spun silk Jute (Note 3) and most other fibres	—
Locust-bean gum and starch	5.4	Cotton (Note 1) Viscose Spun silk	—
Tamarind seed size	5.5	Cotton (Note 1) Viscose, jute	—
Acrylic (size or finish)	5.6	Most fibres (Note 4)	Protein, deacetylated acetate, acetate, triacetate, acrylic, modacrylic
Gelatin and polyvinyl alcohol	5.7	Most fibres	Protein, deacetylated acetate, acetate, triacetate
Starch and polyvinyl alcohol	5.8	Cotton, Polyester	Protein, deacetylated acetate, acetate, triacetate
Polyvinyl acetate	5.9	Most fibres	Deacetylated acetate, acetate, triacetate, chlorofibre
Linseed oil sizes	5.10	Viscose crepe yarns	Protein, deacetylated acetate, acetate, triacetate

(*Continued*)

TABLE 1 APPLICABILITY OF PROCEDURES — *Contd*

NON-FIBROUS MATTER	METHOD	APPLICABLE IN PRESENCE OF	NOT APPLICABLE IN PRESENCE OF
(1)	(2)	(3)	(4)
Amino-formaldehyde resins	5.11	Cotton Regenerated cellulose Deacetylated acetate Acetate Triacetate Polyester Polyamide or Nylon	Asbestos
Bitumen, creosote and tar	5.12	Most fibres	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre
Cellulose ethers	5.13.1	Most fibres	—
—	5.13.2	Cotton	Viscose, deacetylated acetate, triacetate, modacrylic, acrylic
Cellulose nitrate	5.14	Most fibres	Deacetylated acetate, acetate, triacetate chlorofibre
Polyvinyl chloride	5.15	Most fibres	Deacetylated acetate, acetate, triacetate, chlorofibre
Oleates	5.16	Most fibres	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre, polyamide or nylon asbestos
Oxides of chromium- iron and copper	5.17	Deacetylated acetate Acetate Triacetate	—
Pentachlorophenyl laurate (PCPL)	5.18	Most fibres	Polyethylene, polypropylene
Polyethylenes	5.19	Most fibres	Polypropylene
Polyurethanes	5.20	Polyamide or Nylon Regenerated cellulose Deacetylated acetate Acetate Triacetate	Polyester, acrylic, modacrylic

Natural rubbers and styrene butadiene, neoprene, nitrille	5.21	Regenerated cellulose Deacetylated acetate Acetate Triacetate Glass	All synthetic fibres
Silicones	5.22	Most fibres	Polyamide or nylon, glass
Tin weighing	5.23	Silk	—
Wax-based waterproof finishes	5.24	Cotton, Protein, Polyester Polyamide or Nylon	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre

NOTE 1 — Grey cotton loses mass when treated by these methods. The loss amounts to approximately 3% of the final oven-dry mass.

NOTE 2 — Linen loses mass when treated by these methods. The loss depends on the types of yarn from which the fabric is produced. Losses are approximately as follows:

Bleached yarns 2%_m, boiled yarns 3 percent and grey yarns 4 percent.

NOTE 3 — Jute loses mass by approximately 0.5 percent when treated by this method.

NOTE 4 — Polyamide or nylon 6.6 may undergo a loss in mass of fibre substance of up to 1 percent when treated by this method. The loss in mass of polyamide or nylon 6 may vary between 1 percent and 3 percent.

4. APPARATUS

4.1 The apparatus required is part of the normal equipment of a chemical laboratory.

5. PROCEDURES FOR REMOVAL OF NON-FIBROUS MATTER

5.0 When the type of finish present on the material is known the applicable procedures as given in **5.1** to **5.24** shall be followed for removal of non-fibrous matter. In case the type of finish is not known, the procedures as given in Appendix A shall be followed. Information regarding the applicability/non-applicability of the procedures as given in **5.1** to **5.24** in the presence of certain fibres is given in Table 1.

5.1 Oils, Fats and Waxes

5.1.1 Extract the specimen in a Soxhlet apparatus with benzene-methyl alcohol mixture (3 : 2) for two hours at a minimum rate of 6 cycles per hour.

5.2 Soaking Oils

5.2.1 Extract the specimen in a Soxhlet apparatus with toluene-methanol mixture (1 : 3) for two hours at a minimum rate of 6 cycles per hour.

5.3 Starch

5.3.1 Immerse the specimen in a freshly prepared solution containing 0.1 percent by mass of a non-ionic wetting agent together with an appropriate amylase preparation using a liquor/specimen ratio of 100 : 1. The concentration of the amylase preparation and the pH, temperature, and time of treatment should be those recommended by the manufacturer. Transfer the specimen to boiling water and boil it for 15 minutes. Test for complete removal of starch using a dilute aqueous solution of iodine in potassium iodide. When all the starch is removed rinse the specimen thoroughly in water, squeeze or mangle it, and dry it.

5.4 Locust-Bean Gum and Starch

5.4.1 Boil the specimen in water for 5 minutes using a liquor/specimen ratio of 100 : 1. Repeat this procedure with a fresh portion of water. Follow this by the procedure described in **5.3**.

5.5 Tamarind Seed Size

5.5.1 Boil specimen in one percent solution of sodium carbonate for 30 minutes using liquor to material ratio of 30 : 1. Wash it in water and again boil in one percent sodium carbonate solution for 30 minutes. Wash with water and dry.

5.6 Acrylic (Size or Finish)

5.6.1 Immerse and agitate the specimen for 30 minutes in at least 100 times its own mass of solution containing 2 g/l soap or other suitable detergent and 2 g/l sodium hydroxide at 70 to 75°C. Give three 5-minute rinses in distilled water at 85°C, squeeze, mangle or centrifuge, and dry the specimen.

5.7 Gelatin and Polyvinyl Alcohol

5.7.1 Treat the specimen in a solution (using a minimum liquor/specimen ratio of 100 : 1) containing:

- 1 g/l of non-ionic surfactant
- 1 g/l of anionic surfactant, and
- 1 g/l of anhydrous sodium carbonate

for 90 minutes at 50°C followed by 90 minutes in the same bath at 70 to 75°C. Wash the specimen and dry it.

5.8 Starch and Polyvinyl Alcohol

5.8.1 Conduct the procedure described in 5.5 followed by the procedure described in 5.7 with intermediate drying.

5.9 Polyvinyl Acetate

5.9.1 Extract the specimen in a Soxhlet apparatus with acetone for 3 hours at a minimum rate of 6 cycles per hour.

5.10 Linseed Oil Sizes

5.10.1 Conduct the procedure described in 5.1 followed by the procedure described in 5.7.

5.11 Amino-Formaldehyde Resins

5.11.1 Extract the specimen with a solution of 25 g/l ortho-phosphoric acid 50 percent and 50 g/l urea at 80°C for 10 minutes using a liquor/specimen ratio of 100 : 1. Wash the specimen in water, drain, wash it in a 0.1 percent sodium bicarbonate solution, and finally wash it thoroughly in water.

NOTE — This method causes some damage to cupro, viscose, modal, deacetylated acetate, acetate and triacetate.

5.12 Bitumen, Creosote and Tar

5.12.1 Extract the specimen with dichloromethane (methylene chloride) in a Soxhlet apparatus. The duration of treatment depends on the amount of non-fibrous matter present, and it may be necessary to renew the solvent.

NOTE — Extraction of jute with dichloromethane will remove also the batching oil, which may be present to the extent of 5 percent or more.

5.13 Cellulose Ethers

5.13.1 Methyl Cellulose in Cold Water — Soak the specimen in cold water for 2 hours. Rinse the specimen repeatedly in cold water with vigorous squeezing.

5.13.2 Cellulose Ethers Insoluble in Water But Soluble in Alkali — Immerse the specimen in a solution containing approximately 175 g/l sodium hydroxide at room temperature, or cooled to a temperature of approximately 5 to 10°C for 30 minutes. Then wash the specimen thoroughly in a fresh portion of reagent, rinse it well in water, neutralize it with approximately 0.1 N acetic acid, rinse it again in water and dry it.

5.14 Cellulose Nitrate

5.14.1 Immerse the specimen in acetone at room temperature for 1 hour using a liquor/specimen ratio of 100 : 1. Drain, wash the specimen in three portions of fresh acetone, and allow the entrained solvent to evaporate.

5.15 Polyvinylchloride

5.15.1 Immerse the specimen in tetrahydrofuran at room temperature for 1 hour using a liquor/specimen ratio of 100 : 1. If necessary, scrape off the softened polyvinylchloride. Drain, wash the specimen in three portions of fresh tetrahydrofuran, drain and allow the entrained solvent to evaporate.

NOTE — Because of risk of explosion, tetrahydrofuran should not be recovered by distillation.

5.16 Oleates

5.16.1 Immerse the specimen in approximately N/5 hydrochloric acid at ambient temperature until it is thoroughly wetted. Wash the specimen well and dry it. Extract the specimen in a Soxhlet apparatus with dichloromethane (methylene chloride) for 1 hour at a minimum rate of 6 cycles per hour.

5.17 Oxides of Chromium, Iron and Copper

5.17.1 Immerse the specimen in a solution containing 14 g/l hydrated oxalic acid at 80°C for 15 minutes using a liquor/specimen ratio of 100 : 1. Wash it thoroughly (any copper present will remain as the colourless oxalate; remove this with 1 percent acetic acid at 40°C for 15 minutes and wash the specimen). Neutralize the specimen with ammonia and wash it thoroughly in water. Squeeze, mangle or centrifuge, and dry it.

5.18 Pentachlorophenyl Laurate (PCPL)

5.18.1 Extract the specimen in a Soxhlet apparatus with toluene for 4 hours at a minimum rate of 6 cycles per hour.

NOTE — This method is not applicable if dyes containing chromium have been applied to the material under test.

5.19 Polyethylenes

5.19.1 Extract the specimen in boiling toluene under reflux for two hours.

NOTE — The material must be completely immersed in the boiling solvent.

5.20 Polyurethanes

5.20.1 No completely satisfactory method is available but the following have been found useful. Some polyurethanes can be removed by solution in dimethyl sulphoxide or dichloromethane (methylene chloride), and subsequent repeated washing of the sample with fresh quantities of solvent. When the fibre composition of the specimen permits, some polyurethanes can be removed by hydrolysis in an aqueous solution containing 50 g/l sodium hydroxide at the boil for one hour. Alternatively, an aqueous solution containing 50 g/l sodium hydroxide and 100 g/l ethanol may be used at a temperature 50 to 60°C for one hour.

NOTE — Dimethyl sulphoxide has toxic properties.

5.21 Natural, Rubbers Styrene-Butadiene, Neoprene, Nitrile and Most Other Synthetic Rubbers

5.21.1 No completely satisfactory method is available but the following have been found useful.

5.21.2 Soak the specimen in a hot volatile solvent which swells it considerably (for example benzene), and when it is fully swollen remove as much of the rubber as possible by scraping. It may be possible in some cases where the textile fibres are exposed, to wet only the rubber/textile interface, and strip the rubber textile layers apart almost at once.

Continue by boiling the residual specimen with constant stirring in 50 or more times its mass of molten *p*-dichlorobenzene; use a flat-bottomed flask with an attached widebore condenser (to allow adequate access of air), and preferably a magnetic stirrer and hotplate.

5.21.3 After 45 minutes add 1 part 70 percent tri-butyle hydroperoxide per 4 parts *p*-dichlorobenzene present. Boil until decomposition of the rubber is complete (2 hours is an average time). Cool the flask to about 60°C and add an equal volume of benzene. Filter and wash the textile component repeatedly in warm benzene.

5.21.4 Nitrile rubber (i.e. acrylonitrile-butadiene rubber) may require the addition of the same volume of nitrobenzene as of tributyle hydroperoxide to speed up the dissolution process.

NOTE 1 — Natural rubber should dissolve after being boiled in *p*-dichlorobenzene alone for several hours in the presence of air. Solution may also be effected by heating in diphenyl ether at 105°C for 2 hours and then washing the specimen in benzene.

NOTE 2 — The above treatments are strongly oxidative in character and the properties of the textile material may be affected appreciably.

5.22 Silicones

5.22.1 Scour the specimen in a solution containing 50 to 60 millilitre per litre 40 percent hydrofluoric acid in a polyethylene vessel at 65°C for 45 minutes. Thoroughly wash the specimen, neutralize it, and scour it in a solution containing 2 g/l soap at 60°C for 1 hour.

NOTE — Hydrofluoric acid is a dangerous product.

5.23 Tin Weighting (Silk)

5.23.1 Immerse the specimen in N/2 hydrofluoric acid in a polyethylene vessel at 55°C for 20 min and stir occasionally. Rinse in warm water. Immerse the specimen in 2 percent solution of carbonate at 55°C for 20 minutes. Wash the specimen in warm water, squeeze, mangle or centrifuge, and dry it.

5.24 Wax-Based Waterproof Finishes

5.24.1 Extract the specimen in a Soxhlet apparatus with dichloromethane (methylene chloride) for at least 3 hours at a minimum of 6 cycles per hour. Then, to remove any metallic complexes, scour the specimen in a solution containing 10 g/l formic acid and 5 g/l acid-stable surfactant at 80°C for 15 minutes. Wash the specimen thoroughly in water until it is free from acid.

APPENDIX A*(Clause 5.0)*

A-1. Extract the specimens/samples with benzene: methyl alcohol mixture in 3:2 ratio in a Soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes oils, fats, waxes, certain thermoplastic resins, etc).

A-2. Extract the specimens/samples with ethyl alcohol in a Soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes soaps, cationic finishes, etc).

A-3. Treat the specimens/samples with 200 ml of water at 50°C for 30 minutes, stirring occasionally with glass rod or mechanically. Rinse thrice with fresh portion of warm water (50°C) and dry. (This removes water-soluble materials.)

A-4. Immerse the specimen/sample in 200 ml of 0.1 N hydrochloric acid at 80°C for 25 minutes, stirring gently every 3 minutes. Rinse thoroughly with water at 80°C containing a few drops of ammonia and then finally with plain water. Remove excess water from the sample by squeezing, suction centrifuging and allow to dry. (This removes starches/amino-aldehyde compound resins.)

INDIAN STANDARDS

ON

CHEMICAL METHODS OF TEST FOR QUANTITATIVE CHEMICAL ANALYSIS OF FIBRE MIXTURES

IS:

- 667-1955 Simple methods for identification of common commercial textile fibres
- 1564-1962 Method for quantitative chemical analysis of binary mixtures of cellulose triacetate and certain other fibres
- 1889 Method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton:
 - (Part I)-1976 Sodium zincate method
 - (Part II)-1976 Cadoxen solvent method
- 2005-1962 Method for quantitative chemical analysis of binary mixtures of polyamide fibres and certain other fibres
- 2006-1978 Method for quantitative chemical analysis of binary mixtures of protein fibres and certain other fibres (*first revision*)
- 2176-1962 Method for quantitative chemical analysis of binary mixtures of secondary cellulose acetate and certain other fibres
- 2177-1962 Method for quantitative chemical analysis of mixtures of cellulose triacetate and secondary cellulose acetate fibres
- 2727-1964 Method for quantitative chemical analysis of binary mixtures of manila and sisal fibres
- 3416-1966 Method for quantitative chemical analysis of mixtures of polyester fibres with cotton or regenerated cellulose
- 3421-1966 Method for quantitative chemical analysis of binary mixtures of acrylic and certain other fibres
- 6503-1972 Method for quantitative chemical analysis of ternary mixtures of protein fibres, nylon 6 or nylon 6'6, and certain other fibres
- 6504-1972 Method for quantitative chemical analysis of ternary mixtures of viscose rayon, cotton and protein fibres
- 6570-1972 Method for quantitative chemical analysis of binary mixtures of jute and animal fibres